CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 8 September 2006

Crystal Structure Analysis of:

TA26

(shown below)

For Investigator: Theo Agapie ext. 6576

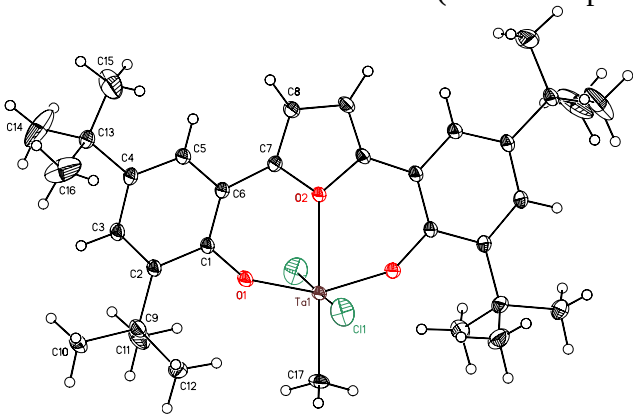
Advisor: J. E. Bercaw ext. 6575

Account Number: JEB.45215-1-ENDOW.452150

By Michael W. Day 116 Beckman ext. 2734

e-mail: mikeday@caltech.edu

Contents



TA26

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 620252. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 620252."

Table 1. Crystal data and structure refinement for TA26 (CCDC 620252).

Empirical formula	$C_{33}H_{45}O_3Cl_2Ta$
Formula weight	741.54
Crystallization Solvent	Not given
Crystal Habit	Block
Crystal size	0.37 x 0.22 x 0.19 mm^3
Crystal color	Dichroic - colorless/red

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 22480 reflections used in lattice determination	2.74 to 42.03°
Unit cell dimensions	a = 29.4428(9) Å b = 10.8239(3) Å β= 98.4960(10)° c = 10.4843(3) Å
Volume	3304.53(17) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	C2/c
Density (calculated)	1.491 Mg/m^3
F(000)	1496
Data collection program	Bruker SMART v5.630
θ range for data collection	2.01 to 42.84°
Completeness to θ = 42.84°	89.4 %
Index ranges	$-55 \le h \le 54$, $-19 \le k \le 19$, $-16 \le l \le 19$
Data collection scan type	ω scans at 5 ϕ settings
Data reduction program	Bruker SAINT v6.45A
Reflections collected	38878
Independent reflections	10832 [R_{int}= 0.0870]
Absorption coefficient	3.518 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.5546 and 0.3560

Table 1 (cont.)

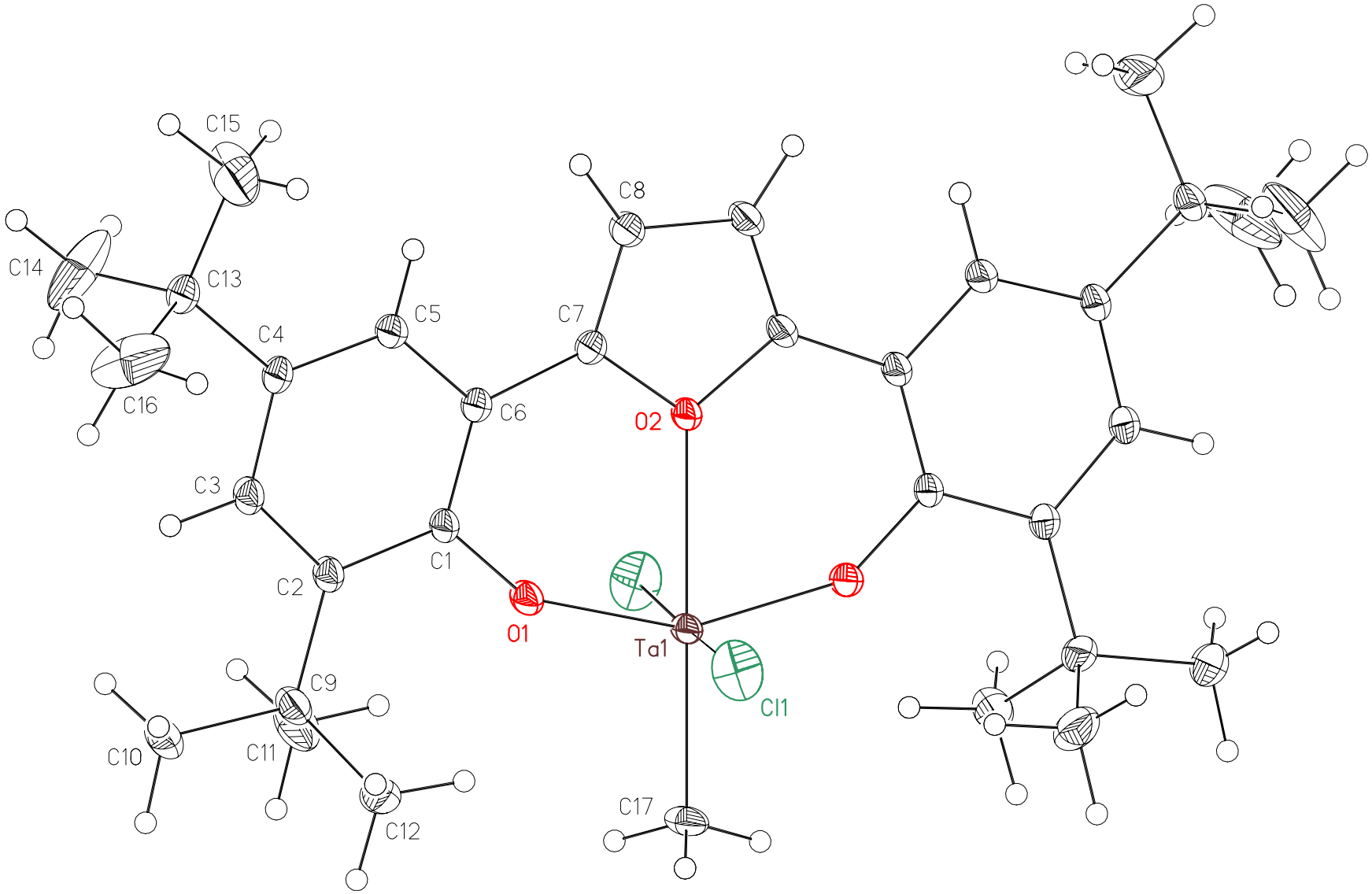
Structure solution and Refinement

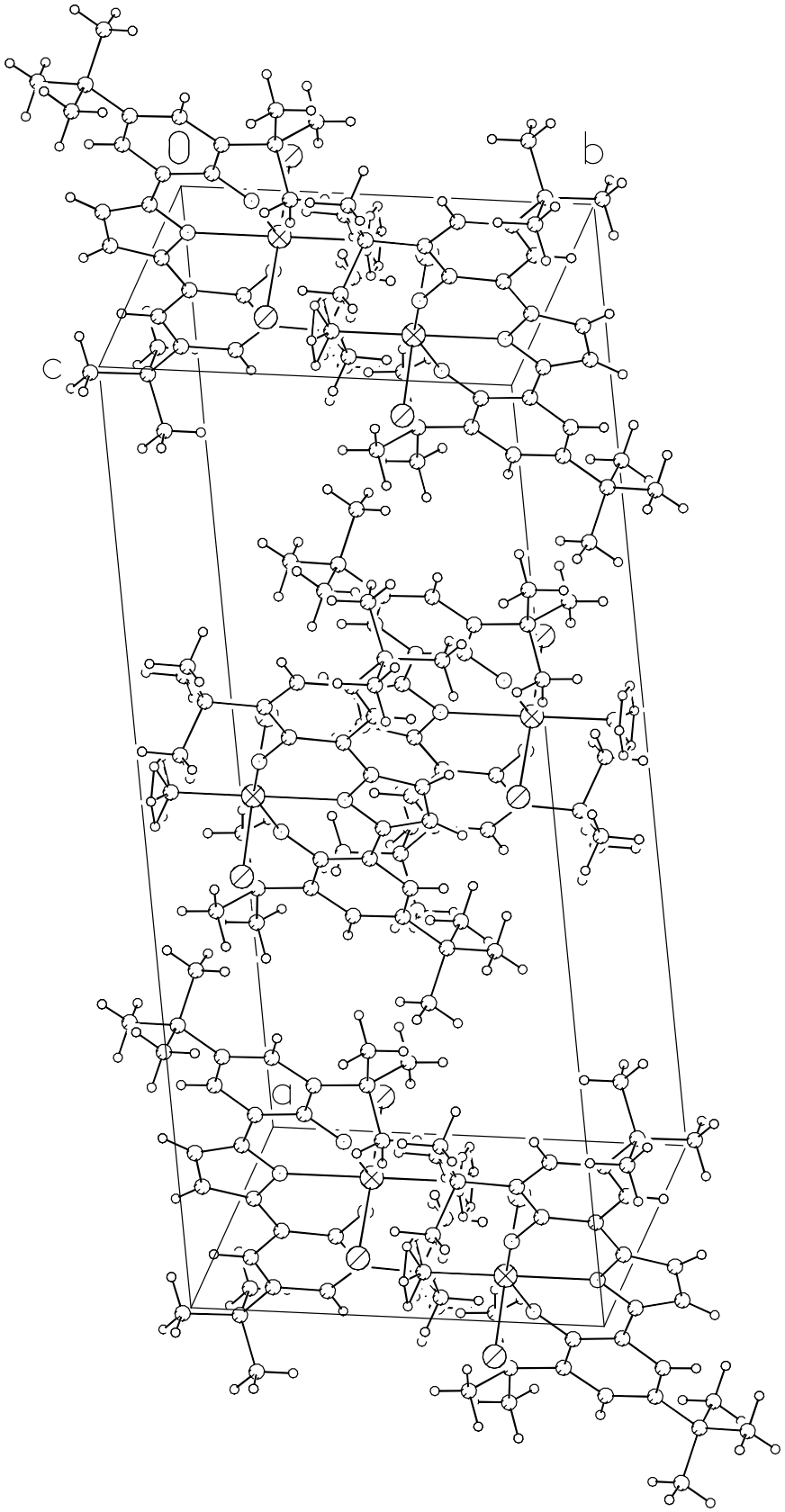
Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	10832 / 0 / 185
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.303
Final R indices [$I>2\sigma(I)$, 8699 reflections]	R1 = 0.0349, *w*R2 = 0.0705
R indices (all data)	R1 = 0.0508, *w*R2 = 0.0732
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.001
Average shift/error	0.000
Largest diff. peak and hole	2.938 and -3.006 e.Å^{-3}

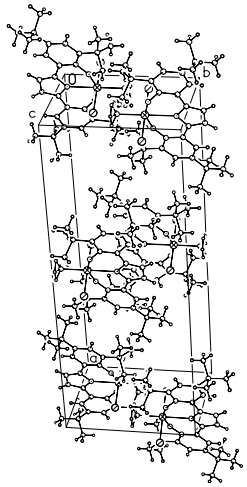
Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.


C15
C14
C13
C16
C4
C5
C8
C7
C6
O2
C3
C1
C2
O1
Ta1
Cl1
C9
C10
C11
C12
C17


O
b
c
a


c
b

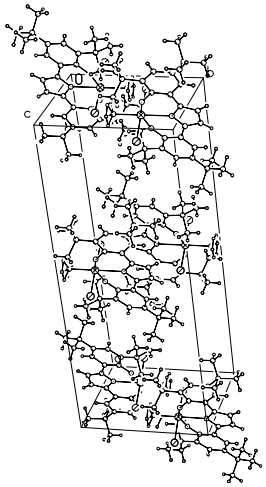

c
b

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for TA26 (CCDC 620252). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Ta(1)	0	2879(1)	2500	14(1)
Cl(1)	466(1)	2849(1)	4538(1)	34(1)
O(1)	517(1)	2467(1)	1746(1)	18(1)
O(2)	0	657(2)	2500	17(1)
C(1)	838(1)	1629(2)	1515(2)	14(1)
C(2)	1240(1)	2043(2)	1049(2)	15(1)
C(3)	1557(1)	1137(2)	841(2)	16(1)
C(4)	1498(1)	-124(2)	1075(2)	16(1)
C(5)	1100(1)	-488(2)	1528(2)	18(1)
C(6)	761(1)	378(2)	1744(2)	16(1)
C(7)	349(1)	-120(2)	2162(2)	19(1)
C(8)	218(1)	-1311(2)	2293(3)	31(1)
C(9)	1320(1)	3408(2)	793(2)	20(1)
C(10)	1782(1)	3630(2)	303(2)	29(1)
C(11)	940(1)	3890(2)	-250(2)	31(1)
C(12)	1336(1)	4162(2)	2044(2)	28(1)
C(13)	1864(1)	-1062(2)	803(2)	18(1)
C(14)	1861(1)	-1109(4)	-653(3)	58(1)
C(15)	1781(1)	-2338(3)	1280(5)	60(1)
C(16)	2338(1)	-638(3)	1427(4)	58(1)
C(17)	0	4869(2)	2500	20(1)

Table 3. Selected bond lengths [Å] and angles [°] for TA26 (CCDC 620252).

Ta(1)-O(1)	1.8692(12)	O(1)-Ta(1)-O(1)#1	152.34(9)
Ta(1)-O(1)#1	1.8692(12)	O(1)-Ta(1)-C(17)	103.83(4)
Ta(1)-C(17)	2.154(2)	O(1)#1-Ta(1)-C(17)	103.83(4)
Ta(1)-Cl(1)#1	2.3626(6)	O(1)-Ta(1)-Cl(1)#1	90.83(5)
Ta(1)-Cl(1)	2.3626(6)	O(1)#1-Ta(1)-Cl(1)#1	88.79(5)
Ta(1)-O(2)	2.4052(18)	C(17)-Ta(1)-Cl(1)#1	90.798(16)
		O(1)-Ta(1)-Cl(1)	88.79(5)
		O(1)#1-Ta(1)-Cl(1)	90.83(5)
		C(17)-Ta(1)-Cl(1)	90.798(16)
		Cl(1)#1-Ta(1)-Cl(1)	178.40(3)
		O(1)-Ta(1)-O(2)	76.17(4)
		O(1)#1-Ta(1)-O(2)	76.17(4)
		C(17)-Ta(1)-O(2)	180.0
		Cl(1)#1-Ta(1)-O(2)	89.202(16)
		Cl(1)-Ta(1)-O(2)	89.202(16)

Symmetry transformations used to generate equivalent atoms:
#1 -x,y,-z+1/2

Table 4. Bond lengths [Å] and angles [°] for TA26 (CCDC 620252).

Ta(1)-O(1)	1.8692(12)	O(1)#1-Ta(1)-O(2)	76.17(4)
Ta(1)-O(1)#1	1.8692(12)	C(17)-Ta(1)-O(2)	180.0
Ta(1)-C(17)	2.154(2)	Cl(1)#1-Ta(1)-O(2)	89.202(16)
Ta(1)-Cl(1)#1	2.3626(6)	Cl(1)-Ta(1)-O(2)	89.202(16)
Ta(1)-Cl(1)	2.3626(6)	C(1)-O(1)-Ta(1)	150.10(12)
Ta(1)-O(2)	2.4052(18)	C(7)-O(2)-C(7)#1	106.95(19)
O(1)-C(1)	1.357(2)	C(7)-O(2)-Ta(1)	126.53(9)
O(2)-C(7)	1.4127(19)	C(7)#1-O(2)-Ta(1)	126.52(9)
O(2)-C(7)#1	1.4128(19)	O(1)-C(1)-C(6)	118.83(14)
C(1)-C(6)	1.399(2)	O(1)-C(1)-C(2)	119.26(15)
C(1)-C(2)	1.418(2)	C(6)-C(1)-C(2)	121.91(14)
C(2)-C(3)	1.394(2)	C(3)-C(2)-C(1)	116.37(15)
C(2)-C(9)	1.527(3)	C(3)-C(2)-C(9)	121.89(14)
C(3)-C(4)	1.402(2)	C(1)-C(2)-C(9)	121.73(14)
C(4)-C(5)	1.385(2)	C(2)-C(3)-C(4)	123.68(14)
C(4)-C(13)	1.537(2)	C(5)-C(4)-C(3)	117.99(15)
C(5)-C(6)	1.410(2)	C(5)-C(4)-C(13)	121.88(16)
C(6)-C(7)	1.453(2)	C(3)-C(4)-C(13)	120.12(14)
C(7)-C(8)	1.359(3)	C(4)-C(5)-C(6)	121.37(16)
C(8)-C(8)#1	1.417(3)	C(1)-C(6)-C(5)	118.67(14)
C(9)-C(11)	1.536(3)	C(1)-C(6)-C(7)	124.93(15)
C(9)-C(12)	1.540(3)	C(5)-C(6)-C(7)	116.38(16)
C(9)-C(10)	1.542(2)	C(8)-C(7)-O(2)	108.22(14)
C(13)-C(15)	1.502(3)	C(8)-C(7)-C(6)	130.04(16)
C(13)-C(16)	1.522(3)	O(2)-C(7)-C(6)	121.74(15)
C(13)-C(14)	1.527(3)	C(7)-C(8)-C(8)#1	108.30(10)
		C(2)-C(9)-C(11)	109.75(16)
O(1)-Ta(1)-O(1)#1	152.34(9)	C(2)-C(9)-C(12)	110.44(16)
O(1)-Ta(1)-C(17)	103.83(4)	C(11)-C(9)-C(12)	110.69(17)
O(1)#1-Ta(1)-C(17)	103.83(4)	C(2)-C(9)-C(10)	112.15(15)
O(1)-Ta(1)-Cl(1)#1	90.83(5)	C(11)-C(9)-C(10)	107.19(17)
O(1)#1-Ta(1)-Cl(1)#1	88.79(5)	C(12)-C(9)-C(10)	106.54(16)
C(17)-Ta(1)-Cl(1)#1	90.798(16)	C(15)-C(13)-C(16)	108.6(3)
O(1)-Ta(1)-Cl(1)	88.79(5)	C(15)-C(13)-C(14)	109.0(3)
O(1)#1-Ta(1)-Cl(1)	90.83(5)	C(16)-C(13)-C(14)	108.1(2)
C(17)-Ta(1)-Cl(1)	90.798(16)	C(15)-C(13)-C(4)	113.06(16)
Cl(1)#1-Ta(1)-Cl(1)	178.40(3)	C(16)-C(13)-C(4)	110.21(17)
O(1)-Ta(1)-O(2)	76.17(4)	C(14)-C(13)-C(4)	107.82(16)

Symmetry transformations used to generate equivalent atoms:
#1 -x,y,-z+1/2

Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for TA26 (CCDC 620252). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ta(1)	92(1)	107(1)	218(1)	0	44(1)	0
Cl(1)	257(2)	455(3)	281(2)	50(2)	-14(2)	-6(2)
O(1)	129(5)	138(5)	276(7)	18(5)	80(4)	4(4)
O(2)	135(7)	123(7)	259(9)	0	97(6)	0
C(1)	113(6)	143(7)	188(7)	5(6)	56(5)	4(5)
C(2)	114(6)	161(7)	173(7)	5(6)	39(4)	-1(5)
C(3)	113(6)	181(7)	188(7)	5(6)	47(5)	3(5)
C(4)	121(6)	173(7)	185(7)	-5(6)	50(5)	21(5)
C(5)	152(6)	138(6)	251(8)	2(6)	84(5)	7(5)
C(6)	131(6)	145(6)	227(8)	-7(6)	76(5)	9(5)
C(7)	169(7)	133(6)	308(9)	-4(6)	129(6)	24(5)
C(8)	284(9)	129(7)	596(15)	-5(9)	300(10)	17(7)
C(9)	162(7)	170(8)	285(9)	39(7)	83(6)	-11(5)
C(10)	229(8)	222(8)	440(12)	32(9)	169(8)	-38(7)
C(11)	248(9)	304(11)	392(12)	182(9)	87(8)	47(7)
C(12)	232(8)	191(8)	433(12)	-87(8)	146(8)	-63(7)
C(13)	131(6)	186(7)	236(8)	-4(6)	62(5)	31(5)
C(14)	669(19)	720(20)	340(13)	-91(14)	77(12)	468(18)
C(15)	462(16)	329(14)	1140(30)	318(17)	520(20)	239(12)
C(16)	146(9)	447(16)	1110(30)	-340(18)	-51(12)	85(9)
C(17)	127(9)	102(8)	384(15)	0	42(8)	0